

## Claim

A method for production of Nd-Fe based plastic magnet material characterized by subjecting a powder obtained by pulverizing an Nd-Fe based alloy ingot in a chemical treatment to remove the surface layer of the powder particles followed by a heat treatment in a non-oxidizing atmosphere at a temperature of 500°C or above, adding a plastic binder to mix, and further subjecting to compression molding or injection molding.

## Detailed explanation of the invention

### Industrial application field

The present invention pertains to a method for production of an Nd-Fe based plastic magnet material.

### Prior art

Permanent magnet materials are widely used for home electrical appliances, precision machines and automobile parts and, with the increased demand for reduced size and increased performance of electrical and electronic equipment, further improvement in magnetic performance is required.

Many suggestions have been made for the use of Nd-Fe based plastic magnet material, as well, and for production of plastic magnet materials, a method consisting of quenching and coalescing an alloy to form a ribbon, crushing the material to form a powder with a particle size of approximately 200  $\mu$  and mixing with a plastic, and molding to form an isotropic plastic magnet material, and a method consisting of melting an alloy ingot followed by a heat treatment, then, crushing the material to form a fine powder and mixing the powder with a plastic and subjecting to compression molding or injection molding are known methods.

### Problems to be solved by the invention

However, the reduction in the coercive force is significant in the plastic magnet material obtained by the latter method, that is, the plastic magnet material obtained by crushing an alloy ingot and mixing with a plastic followed by molding; thus, it is not possible to produce a permanent magnet material suitable for actual application.

The present invention is based on the aforementioned problems of the prior art. And as such, the purpose of the present invention is to provide a method for production of a plastic magnet material with superior magnetic properties using a fine powder obtained by pulverizing an Nd-Fe based alloy ingot.

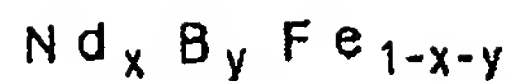
### Means to solve the problem

As a result of much research conducted by the inventors of the present application, the inventors discovered that the aforementioned purpose could be achieved when a chemical treatment is provided for a fine powder obtained by pulverizing an Nd-Fe based ingot by mechanical means, followed by a heat treatment, and as a result, the present invention was accomplished.

Thus, the method for production of a Nd-Fe based plastic magnet material of the present invention is characterized by subjecting a powder obtained by pulverizing an Nd-Fe based alloy ingot to a chemical treatment to remove the surface layer of the particle powder, followed by a heat treatment in a non-oxidizing atmosphere at a temperature of 500°C or above, adding a plastic binder to mix, and further subjecting to a compression molding or injection molding.

The present invention is explained in further detail below.

For an Nd-Fe based alloy ingot used in the present invention, those represented by the general formula below can be used effectively.



(In the formula,  $0.05 \leq x \leq 0.30$ ,  $0.01 \leq y \leq 0.1$  (molar ratio))

In the aforementioned general formula, a part of the Nd may be replaced with other rare earth elements, furthermore, a part of B may be replaced with one or a plurality of elements selected among C, N, Si, P or Al, as well. Furthermore, up to 20 wt% of the Fe may be replaced with one or a plurality of elements selected from Co, Mn, Ni, Ti, Zr, Hf, V, Nb, Cr, Ta, Mo and W, as well.

Pulverization of the aforementioned Nd-Fe based alloy ingot can be completed using a standard method. For example, first, coarse crushing is performed by a jaw crusher, then, medium crushing is provided by a disc crusher, and finally, fine grinding is achieved by a jet mill to form particles with a particle diameter in the range of several microns.

The fine powder obtained as a result of pulverization is subsequently subjected to a chemical treatment to remove the surface layer of the particle. For example, a chemical treatment consisting of dipping the fine powder in an acid solution for a short time may be used. For the acid used in this case, inorganic acids, for example, nitric acid or a mixture of nitric acid and sulfuric acid, etc., can be mentioned, and it is desirable when used as a solution obtained by adding to an alcohol.

The fine powder subjected to a chemical treatment is further subjected to a heat treatment in a non-oxidizing atmosphere at a temperature of 500°C or above, and for the non-oxidizing atmosphere, hydrogen gas or an inert gas, for example, argon, nitrogen, etc., can be mentioned, and use of hydrogen gas is especially desirable in this case.

After the aforementioned heat treatment, the fine powder may be isolated by a ball mill, etc., a plastic is added as a binder, and the combination is mixed. The mixture obtained is subjected to compression molding or injection molding to give an Nd-Fe based plastic magnet material having a specific shape. For the plastic in this case, known plastics may be used, and for example, in the case of compression

molding, curable resins such as epoxy resins and phenol resins are used, and in the case of injection molding, polyamides such as nylons, polyolefins such as propylene, polyesters such as polyethylene terephthalate, etc., can be used successfully.

Furthermore, it is desirable when the aforementioned compression molding or injection molding is performed in a magnetic field to give an anisotropic plastic magnet material.

#### Function

In the present invention, the Nd-Fe based alloy ingot is pulverized, a chemical treatment is further provided for the fine powder obtained and the surface layer of the particles of the fine powder is removed, and as a result, removal of the distortions and oxide existing on the surface of the particles formed at the time of the pulverization of the alloy ingot is made possible. Furthermore, after the aforementioned treatment, the fine powder is further subjected to a heat treatment under a non-oxidizing atmosphere; as a result, distortions inside the particle of the fine powder can be eliminated without causing oxidation on the surface of the particles. When the fine powder provided with the aforementioned treatments is used and mixed with a plastic binder followed by a compression molding or injection molding, production of a plastic magnet material having excellent magnetic properties can be achieved.

#### Application example

The present invention is explained in further detail with a working example below.

An alloy having a composition comprised of  $\text{Nd}_{16}\text{Fe}_{68.9}\text{C}_{0.5}\text{B}_{10}\text{N}_{0.1}$  was produced by a button arc furnace in an argon atmosphere. The alloy ingot obtained was subjected to pulverization using a jaw crusher, then, a disc crusher, and finally, a jet mill to give a fine powder having a mean particle diameter

of 5  $\mu\text{m}$ . The aforementioned fine powder was dipped in a 1% nital (1 vol% of 40% nitric acid + balance ethyl alcohol to form 100%) for 10 sec. Subsequently, the powder was washed in ethyl alcohol, then dried. Then, a heat treatment was applied to the powder in a non-oxidizing atmosphere for 1 h at a temperature of 600°C. Furthermore, 2 wt% of epoxy resin was added to mix. Then, the mixture obtained was compression molded in a magnetic field of 15 KOe under a pressure of 7 ton/cm<sup>2</sup>. The properties of the plastic magnet material obtained are shown in the table below.

Furthermore, for comparison, the magnetic properties of a plastic magnet material sample produced without providing a treatment with nital, a sample produced without providing a heat treatment and a sample produced without either of the aforementioned treatments are also shown in the table.

		① 磁 気 特 性			
		B <sub>r</sub> (G)	B <sub>Hc</sub> (Oe)	H <sub>c</sub> (Oe)	(BH) <sub>max</sub> (MG · Oe)
②	ナイタル処理有り	6100	5000	10500	0.3
	熱処理無し ③	3000	1000	15000	1.2
④	ナイタル処理無し	2800	880	1300	0.5
	無処理 ⑤	2800	860	1200	0.4

- Key: 1      Magnetic properties
- 2      Nital treatment provided
- 3      Absence of heat treatment
- 4      Absence of nital treatment
- 5      Heat treatment

#### Effect of the invention

According to the present invention, a chemical treatment is provided for a fine powder obtained by pulverizing an Nd-Fe based alloy ingot and treating with an acid, followed by a heat treatment in a

non-oxidizing atmosphere; thus, distortion caused by pulverizing and oxide is thoroughly removed from the surface of the particles of the fine powder, and so, the plastic magnet material molded from the aforementioned fine powder exhibits excellent magnetic properties, as is clearly shown in the table above.